

**FIG. 1**

**PROVIDE AN ALUMINA POWDER PRECURSOR**

**ADD ABOUT 1-10% MAGNESIA POWDER  
PRECURSOR AND 1-10% TITANIA POWDER  
PRECURSOR TO THE ALUMINA POWDER  
PRECURSOR TO MAKE A GREEN POWDER  
PRECURSOR**

**MIX THE GREEN POWDER PRECURSOR**

**PRESSING A GREEN BODY FROM THE GREEN  
POWDER PRECURSOR**

**REMOVING RESIDUAL MOISTURE AND  
ORGANIC MATERIAL FROM THE GREEN BODY**

**FIRING THE GREEN BODY IN AIR TO ABOUT  
CONE 13**

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**FIGURE 2**

**Some experimental data for a typical low-fired high alumina body**

Alumina Content	Low Firing Alumina Composition
Acid Resistant	Silica-Free Formulation
Starting Grain Size	$\frac{1}{2} \mu\text{m}$
Grain Growth	1-3 $\mu\text{m}$ (avg. 1.2 +/- 0.3 $\mu\text{m}$ )
Material Preparation	Ball Milling, Spray Drying, Dry Pressing
Firing	Performed in Normal Atmosphere, Fiber-Lined Furnace, Fired to Conc 13
Specific Gravity	3.8 + (@ 1350°C)
Water Absorption	0
Thermal Conductivity @ 25°C (cal/cm <sup>2</sup> /sec/°C)	0.05
Thermal Expansion @300 - 1000°C (°C)	$8.3 \times 10^{-6}$
Thermal Expansion Rate	Substantially Uniform – There is Negligible Thermal Expansion Mismatch Between the Matrix and Second Phase
Toughness, K <sub>IC</sub>	4-5 Mpa-m <sup>1/2</sup>
Rockwell Hardness (45N)	>80
Hardness (GPa)	16
Elastic Modulus	365 Gpa
Flexural Strength (kpsi)	47
Compressive Strength (kpsi)	>300
Tensile Strength (kpsi)	25
Dielectric Strength (v/mil)	250 (open atmosphere)
Dielectric Constant @ 1 kHz	8.2
Dissipation Factor @ 1 kHz	0.001
Loss Factor @ 1 kHz	.01
T <sub>e</sub> (°C)	>900
Volume Resistivity @ 25°C	>10 <sup>14</sup>
Surface Finish as Fired:	10 rms ± 2 (typical sample)
Ground:	5 rms ± 1 (typical sample)

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**FIGURE 3**

**Long-Term Corrosion Study of Typical Low-Temperature High-Alumina (LTHA):**

<u>MATERIAL</u>	<u>WEIGHT LOSS</u>
ZTA	-70 percent
SiC/SiC	-30 percent
LTHA Sample	-30 percent

**Materials Corrosion Test:  
Independent Test**

**Weight loss in mg/dm<sup>2</sup>/day**

46.7% Hydrofluoric (HF) acid @ 25°C

	<u>5 day immersion</u>	<u>35 day immersion</u> (30 after 5)
SiC-Silica Free	1.00	1.00
ZrO <sub>2</sub> -Toughened	1110.00	1070.00 <sup>(1)</sup>
Al <sub>2</sub> O <sub>3</sub> – 99.9%	1.92	2.26
<b>LTHA Sample</b> (Membrane-approx. 36% porosity)	1.0	0.16 <sup>(2)</sup>
<b>LTHA Sample</b> (Solid)	1.64	0.09

**NOTE:** Weight loss is listed in mg/dm<sup>2</sup>/day rounded to nearest 0.01g.

- (1): Approximately 2/3 of the coupon was destroyed in 35 days of testing.  
(2): This is a rather severe test in that the surface area is approx. 36% greater than the normal as tested.

**Materials Corrosion Test:**

50% H<sub>3</sub>PO<sub>4</sub> @ 25°C

	<u>Cum. Mg/dm<sup>2</sup> (approx.)</u>	
	24 Hours	120 Hours
AD90	5.35	9.65
AD94	2.72	5.00
AD96	4.82	12.54
ADO96	5.61	11.59
AD99.5	6.75	10.26
TTZ	0.88	3.33
LTHA Sample	1.66	2.02



30% NaOH @ 25°C

Cum. Mg/dm<sup>2</sup> (approx.)

	24 Hours	120 Hours
AD90	24.98	51.15
AD94	15.24	32.27
AD96	2.13	6.10
ADO96	11.59	14.61
AD99.5	8.23	12.20
TTZ	0.61	0.61
LTHA Sample	1.72	2.01

**NOTE:** Weight loss is mg/dm<sup>2</sup>/day, rounded to nearest 0.01g.

**Materials Corrosion Test:**

Weight loss in mg/cm<sup>2</sup>/day

	<u>60% H<sub>3</sub>PO<sub>4</sub> @ 60°C</u>	<u>30% NaOH @ 60°C</u>
A479 Al <sub>2</sub> O <sub>3</sub> (90%)	0.15	0.28
A479SS Al <sub>2</sub> O <sub>3</sub> (99.5%)	0.07	0.12
3NaI <sub>2</sub> O <sub>3</sub> (99.9%)	0.02	0.00
LTHA Sample	0.00	0.00

**NOTE:** Weight loss is mg/cm<sup>2</sup>/day, rounded to nearest 0.01g.

**High Alumina Corrosion Test:**

Independent Test

<u>CORROSIVE SOLUTION</u>	<u>HCl</u>	<u>HNO<sub>3</sub></u>	<u>H<sub>2</sub>SO<sub>4</sub></u>
<u>MATERIAL</u>	<u>% WEIGHT LOSS</u>		
Product of Manufacturer A:			
85% Al <sub>2</sub> O <sub>3</sub>	0.066	0.076	0.066
96% Al <sub>2</sub> O <sub>3</sub>	0.081	0.087	0.200
LTHA Sample	(No Detectable Loss)		
Product of Manufacturer B:			
99.5% Al <sub>2</sub> O <sub>3</sub>	0.217	0.163	0.216

**PROCEDURES**

1. Check the initial weight (approximately 5 grams)
2. Immerse into high concentration acid/base solutions
3. Dilute with 50 volume % of distilled water
4. Boil for an hour, and let soak overnight
5. Check the final weight
6. Calculate percent weight loss

$$\% \text{ LOSS} = (\text{INITIAL WEIGHT} - \text{FINAL WEIGHT}) / \text{INITIAL WEIGHT}$$

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**FIGURE 4**

**Summary of Typical Low-Temperature High-Alumina Material Thermal Spray Features:**

		<u>APPLICATION METHOD</u>	
		<u>Subsonic Plasma</u>	<u>HVOF</u>
Deposit Efficiency (DE)		85+%	75+%
Lay-Down Rate (surface speed = 1200 ipm)		4+mil/pass	1+mil/pass
Hardness (as applied)	Rockwell Vickers	90+ 1,100+	94+ 1300+
Surface Roughness (as applied)		200 – 300 $\mu$ in.	60 – 80 $\mu$ in.
Dielectric Strength		450+v/mil	950+v/mil
Bond Strength		10,000 – 12,000 psi	
Porosity (by optical microscopy)	Interconnected Surface		0% < 1/2 $\mu$ in.

**Properties of LTHA Coating**

		<u>APPLICATION METHOD</u>	
		<u>Subsonic Plasma*</u>	<u>HVOF**</u>
Deposit Efficiency (DE)		85.5% (82 - 91)	74% (65 - 83)
Lay-Down Rate (mil/pass) (surface speed = 1200 ipm)		3.5 (2 - 4.2)	0.98 (0.87 – 1.03)
Hardness (as applied)	Rockwell Std. Dev.	90.1 (88.9 – 91.7) 1.4	94.3 (94.1 – 94.7) 0.4
Surface Roughness (as applied)		200 – 300 $\mu$ in.	60 – 80 $\mu$ in.
Dielectric Strength		450+v/mil	950+v/mil
Bond Strength		10,000 – 12,000 psi	
Porosity (by optical microscopy)	Interconnected Surface		0% < 1/2 $\mu$ in.

\* These averages are based on 132 sample coupons, randomly picked for testing out of approximately 30 spray runs containing 8 – 10 coupons per run.

\*\* These averages are based on 30 sample coupons, randomly picked for testing out of approximately 10 spray runs containing 8 – 10 coupons per run.

( ) Indicates ranges of high and low values.

**FIGURE 5****Tensile Test Data for 6061 Matrix Composites at 15v% (Test One)**

(NOTE: This test conducted with non-spherical grain)

Material	H.T. Condition	Elastic Modulus (Msi)	Yield Strength (ksi)	Ultimate Strength (ksi)	Strain at Failure (%)
6061 (avg.)	T - 6	9.9	40	45	10
LTHA Sample	T - 6	12.9	53	65	7
Tabular Al <sub>2</sub> O <sub>3</sub>	T - 6	12.9	53	65	7

**Tensile Test Data for 7093 Matrix Composites at 15v% (Test Two)**

Material	H.T. Condition	Elastic Modulus (Msi)	Yield Strength (ksi)	Ultimate Strength (ksi)	Strain at Failure (%)
7093 (avg.)	T - 6	10.3	92	95.6	13.4
LTHA Sample	T - 6	13.1	80	85	0.9
Tabular Al <sub>2</sub> O <sub>3</sub>	T - 6	13.4	58	67	4.2
B <sub>4</sub> C	T - 6	14.6	84	98	2.6

**Tensile Test Data for 7093 Matrix Composites at 10v% (Test Three)**

Material	H.T. Condition	Elastic Modulus (Msi)	Yield Strength (ksi)	Ultimate Strength (ksi)	Strain at Failure (%)
7093 (avg.)	T - 6	10.3	92	95.6	13.4
LTHA Sample	T - 6	12.2	82.5	90.0	3.7
B <sub>4</sub> C (96 samp.)	T - 6	13.2	89	96.7	4.1

Thermal Expansion Coefficient: 10 ppm/°F (Al = 13 ppm/°F)

**Friction and Water Data:**

Composite ID	Coefficient of Friction		Volume loss from Block (10 <sup>-3</sup> cu cm)
	Start	Finish	
20v% SiC/2124	.096	.119	6.34
25v% SiC/2124	.101	.123	6.23
30v% SiC/2124	.098	.119	4.15
20v% SiC/7091	.101	---	6.31
SPF-251 Std Coating	.141	.129	13.11

**Test Results of 7093/Al<sub>2</sub>O<sub>3</sub>/xyp Composite**

Sample	Vol. %	YS 0.2% (ksi)	UTS (%)	Elongation (Msi)	Modulus
Baseline T - 6	0	85.7	96.5	22.4	10.6
Tabular Al <sub>2</sub> O <sub>3</sub> T - 6	15	58.0	67.0	4.2	14.1
Medialox C25CR	10	25.1	33.9	2.43	---
Duralox DF500	10	36.5	48.7	7.56	---
Baikalo GE6	10	24.9	37.0	4.53	---
LTHA Sample Lot 1 T - 6	15	84.3	86.2	0.9	13.5
LTHA Sample Lot 2 T - 6	10	82.5	89.6	3.7	12.2

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